Data Validation SOP

HW-17, Rev. 1.3

Herbicides

YES NO N/A

This Region II SOP document is based on SW846 Method 8150A, Revision I, July,1992

1.0 Traffic Reports and Laboratory Narrative

1.1 Are Traffic Report Forms present for all samples? [ ] ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or SDG Narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data? ___ [ ]

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be qualified as estimated (J). If a soil sample, other than TCLP, contains more than 90% water, all data should be qualified as unusable (R).

ACTION: If samples were not iced upon receipt at the laboratory, flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any technical holding times,determined from date of collection to date of extraction, been exceeded? ___ [ ]

Note:Water and soil samples for herbicide analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date extraction. However, the SAS Client Request takes precedence and the Holding Times specified in the SAS are the criteria used for validating data.

YES NO N/A

ACTION: If technical holding times are exceeded, flag all positive results as estimated (J) and sample quantitation limits (UJ) and document in the narrative that holding times were exceeded. If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re-analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all the data should at least be qualified "J", but the reviewer may determine that non-detects are unusable (R).

3.0 Surrogate Recovery (Form II)

3.1 Are the Herbicide Surrogate Recovery Summaries (Form II) present for each of the following matrices?

a. Low Water [ ] ___

b. Soil [ ] ___

3.2 Are all the Herbicide samples listed on the appropriate Surrogate Recovery Summary for each of the following matrices?

a. Low Water [ ] ___

b. Soil [ ] ___

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document effect in data assessments.

3.3 Were outliers marked correctly with an asterisk? [ ] ___

ACTION: Circle all outliers in red.

3.4 Were surrogate recoveries outside of the advisory limits for any sample or blank? (50-120%) ___ [ ]

YES NO N/A

ACTION: No qualification is done if the surrogate is diluted out. If recovery for the surrogate is below the contract limit, but above 10%, flag all results for that sample 'J". If recovery is < 10%, qualify positive results 'J" and flag non-detects "R". If recovery is above the contract advisory limit qualify positive values "J".

3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point calibration analysis? (see Form VI Herb-1) [ ] ___

ACTION: If the RT limits are not met, the analysis may be qualified unusable (R) for that sample on the basis of professional judgement.

3.6 Are there any transcription/calculation errors between raw data and Form II? ___ [ ]

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document effect in data assessments.

4.0 Matrix Spikes (Form III)

4.1 Is the Matrix Spike/Matrix Spike Duplicate Recovery Form (Form III) present? [ ] ___

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices? (1 MS/MSD must be performed for every 20 samples of similar matrix or concentration level)

a. Low Water [ ] ___

b. Soil [ ] ___

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above.

YES NO N/A

4.3 How many herbicide spike recoveries are outside QC limits (60-140%)?

Water	Soil
_____ out of _____	_____ out of

4.4 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water	Soil
_____ out of ____	_____ out of

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria and determine the need for some qualification of the data.

5.0 Blanks (Form IV)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___

5.2 Frequency of Analysis: has a reagent/ method blank been analyzed for each SDG or every 20 samples of similar matrix or concentration or each extraction batch, whichever is more frequent? [ ] ___

ACTION: If any blank data are missing, take the action specified above in 3.2. If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

5.3 Has a Herbicide instrument blank been analyzed at the beginning of every analytical sequence of 10 samples ? [ ] ___

YES NO N/A

ACTION: If any blank data are missing, call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in data assessments.

5.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for Herbicides? [ ]

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent/cleanup blanks have positive results for Herbicides? When applied as described in table below, the contaminant concentration in the method blank is multiplied by the sample Dilution Factor and corrected for % moisture when necessary. ___ [ ]

6.2 Do any field/rinse blanks have positive Herbicides results? ___ [ ]

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet)

NOTE: All field blank results associated to a particular qualified because of contamination in another blank. Field blanks must be qualified for surrogate, calibration, or any QC problems.

YES NO N/A

ACTION: Follow the directions in the table below to qualify TCL results due to contamination. Use the largest value from all the associated blanks.

Sample conc > CRQL but < 5x blank	Sample conc < CRQL & is < 5x blank value	Sample conc > CRQL & > 5x blank value
Flag sample result with a "U";	Report CRQL & qualify "U"	No qualification is needed

NOTE in the associated samples should be qualified as unusable (R).

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ]

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

7.0 Calibration and GC Performance

7.1 Are the Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, QC Check reference, and MS/MSD? [ ] ___

ACTION: If no, take action specified in 3.2 above.

7.2 Are Forms VI - Herbicides 1,2,4 present and complete for each column and each analytical sequence? [ ] ___

ACTION: If no, take action specified in 3.2 above.

7.3 Are there any transcription/calculation errors between raw data and Forms VI? ___ [ ]

YES NO N/A

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.

7.4 Were the retention time windows calculated using the average absolute retention time (at least three measurements) ± three times the standard deviation of the absolute retention time, for each standard? (Refer to Method 8000A, section 7.5). [ ] ___

7.5.1 Was a QC check standard analyzed prior to environmental samples? [ ] ___

7.5.2 If yes, was the surrogate recovery >50%? [ ] ___

7.5.3 Was the QC check standard re-extracted/re-analyzed, if surrogate recovery was <50%, or any one analyte was < 40%, or two analytes < 70% ? [ ] ___

Action: If NO to any of the above, then qualify positive hits as estimated "J" and non-detects as rejected "R" in the original analysis of all samples in the associated analytical sequence.

7.6 Do all standard retention times, including each Herbicides in each level of Initial Calibration fall within the windows established during the initial calibration analytical sequence? (For Initial Calibration Standards, Form VI - Herbicides - 1). [ ]

ACTION: If no, all samples in the entire analytical sequence are potentially affected. Check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogate is visible, non-detects are valid. If peaks are present and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

YES NO N/A

7.7 Are the linearity criteria for the Initial Calibration analyses within limits for both columns? (% RSD must be < 20.0% for all analytes). [ ]

ACTION: If no, qualify all associated positive results generated during the entire analytical sequence "J" and all non-detects "UJ". When RSD >90%, flag all non-detect results for that analyte R (unusable).

7.8 Are there any transcription/calculation errors between raw data and Form VII - Herbicides-2? ___ [ ]

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

7.9 Is the resolution between any two adjacent peaks in the QC Reference Check Mixture > 60.0% for both columns? (Form VI-Herbicides- 4) [ ] ___

ACTION: If no, positive results for compounds that were not adequately resolved should be qualified "J". Use professional judgement to determine if non-detects which elute in areas affected by co-eluting peaks should be qualified "N" as presumptive evidence of presence or unusable (R).

7.10 Is Form VII -Continuing Calibration present and complete for each analytical sequence for both columns? [ ] ___

ACTION: If no, take action as specified in 3.2 above.

7.11 Have all samples been injected within a 24 hr. period beginning with the injection of the first standard? [ ] ___

YES NO N/A

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify accordingly.

7.12 Do all analyte retention times for the Mid-concentration Check standard (Form VII Herb-2) fall within the windows established by the initial calibration sequence? [ ] ___

ACTION: If no, beginning with the samples which followed the last in-control standard, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times. If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present

and cannot be identified through pattern recognition or using a revised RT window, qualify all positive results and non-detects as unusable (R).

7.13 Are RPD values for all verification calibration standard compounds < 25.0% [ ] ___

ACTION: The "associated samples" are those which followed the last in-control standard up to the next passing standard containing the analyte which failed the criteria.

If %D is 25 -50% qualify as "J"
If %D is 50-100% qualify as "NJ"
If %D is >100% qualify as "R"
If %D is >100% with visible interferences/qualify as "JN"

8.0 Analytical Sequence Check (Form VIII)

8.1 Is Form VIII present and complete for each column and each period of analyses? [ ] ___

ACTION: If no, take action specified in 3.2 above.

YES NO N/A

8.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? (see SAS Client Request/section 8/paragraph 6) [ ] ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

9.0 Herbicides Identification

9.1 Is Form X complete for every sample in which a Herbicide was detected? [ ] ___

ACTION: If no, take action specified in 3.2 above.

9.2 Are there any transcription/calculation errors between raw data and Form X. ___ [ ]

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note errors in data assessment.

9.3 Are retention times (RT) of sample compounds within the established RT windows for both columns? [ ] ___

Was GC/MS confirmation provided instead of confirmation by a second dissimilar column? [ ] ___

Action: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis or by GC/MS. Also qualify as unusable (R) all positive results not meeting RT window unless associated standard compounds show a similar RT shift. The reviewer should use professional judgement to assign an appropriate quantitation limit.

YES NO N/A

9.4 Is the percent difference (% D) calculated for the positive sample results on the two GC columns < 25.0%? [ ] ___

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be flagged as follows:

% Difference	Qualifier
25-50 %	J
50-90 %	JN
> 90 %	R

NOTE: The lower of the two values is reported on Form I. If using professional judgement, the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

9.5 Check chromatograms for false negatives. Were there any false negatives? ___ [ ]

ACTION: Use professional judgement to decide if the compound should be reported.

10.0 Compound Quantitation and Reported Detection Limits

10.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? ___ [ ]

NOTE: The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interfering compound is indicated, the lower of the two values should be reported and qualified as presumptively present at an approximated quantity (NJ). This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate the presence of interferences during the evaluation of the second column confirmation.

YES NO N/A

10.2 Are the CRQLs adjusted to reflect sample dilutions and, for soils, % moisture? [ ] ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest CRQLs are used (unless a QC exceedance dictates the use of the higher CRQL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: Quantitation limits affected by large, off-scale peaks should be qualified as unusable (R). If the interference is on-scale, the reviewer can provide an approximated quantitation limit (UJ) for each affected compound.

10.3 Have all data (Forms and associated chromatograms and quantitation reports) been submitted for original, diluted or re-extraction/re-analysis samples? [ ] ___

11.0 Chromatogram Quality

11.1 Were baselines stable? [ ] ___

11.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ]

ACTION: Address comments under System Performance of data assessment. Explain use of professional judgement where used to qualify data.

YES NO N/A

12.0 Field Duplicates

12.1 Were any field duplicates submitted for Herbicides analysis? [ ]

Note: Check whether SAS Client Request required field duplicates.

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.